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Sampling and Analysis Experiments for Improved Characterization of Products of Incomplete Combustion

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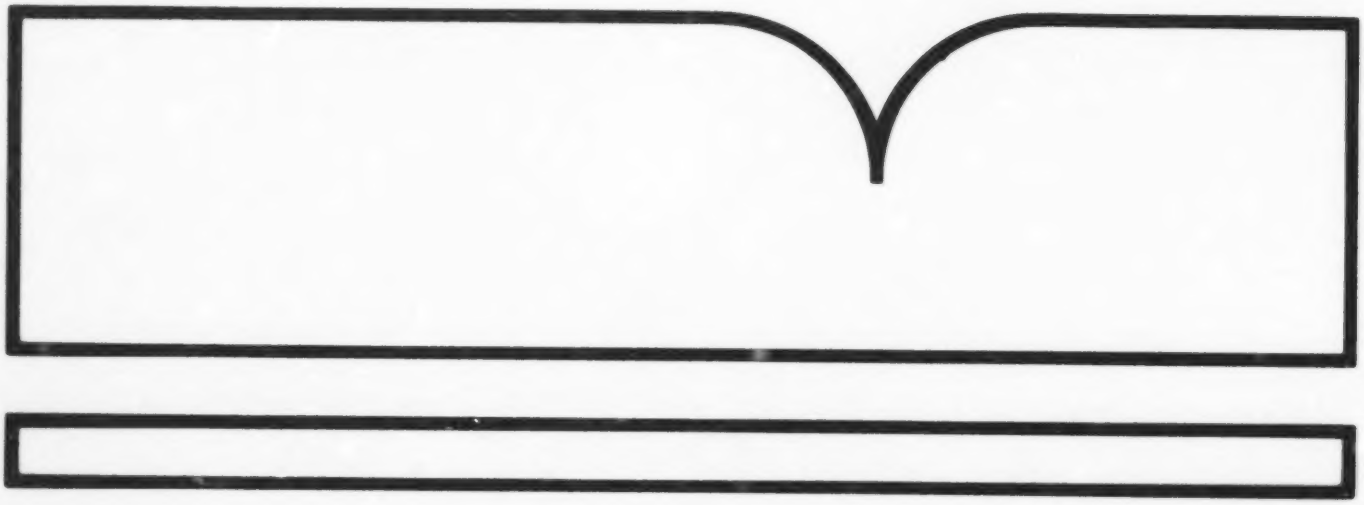

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16. ABSTRACT
Laboratory and field experiments were conducted to evaluate the Volatile Organic Sampling Train (VOST) methodology for the sampling and analysis of products of incomplete combustion (PICs). A pilot-scale incinerator was used to incinerate several volatile chlorinated organic compounds which were known to produce PICs. Approximately 300 ppm of 1,2-dichloroethane, 1,1,1-trichloroethane, and trichloroethene were individually incinerated at 700°C. Standard VOST sampling apparatus was used to collect samples for determinations of precision and for distributive volume studies. A series of experiments was conducted to establish whether PICs were being formed at concentration levels which could be measured by the VOST methodology. Samples were collected at 1 L/min for 20 min with standard pairs of Tenax-Tenax/charcoal cartridges. The major PICs found were vinyl chloride from 1,2-dichloroethane, 1,1-dichloroethene from 1,1,1-trichloroethane, and carbon tetrachloride from trichloroethene. Chloromethane was also formed at significant levels for all of the compounds incinerated. Additional field samples that were collected and analyzed showed that the precision of the PIC generation was fairly constant. Use of a capillary Megabore column, to improve chromatographic resolution, was also evaluated. This modification was used to enhance the Gas Chromatography/Mass Spectrometry performance and allow improved identification of compounds in the field samples. An Environmental Protection Agency audit cylinder containing compounds at approximately 5 ppb was also sampled and analyzed. Results showed these procedures incorporating VOST methodology were accurate and precise.

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Sampling and Analysis Experiments for Improved Characterization of Products of Incomplete Combustion

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Laboratory and field experiments were conducted to evaluate the volatile organic sampling train (VOST) methodology for the sampling and analysis of products of incomplete combustion (PICs). A pilot-scale incinerator was used to incinerate several volatile chlorinated organic compounds which were known to produce PICs. Approximately 300 ppm of 1,2-dichloroethane, 1,1,1-trichloroethane, and trichloroethene were individually incinerated at 700°C. Standard VOST sampling apparatus was used to collect samples for determinations of precision and for distributive volume studies. A series of experiments was conducted to establish whether PICs were being formed at concentration levels which could be measured by the VOST methodology. Samples were collected at 1 L/min for 20 min with standard pairs of Tenax-Tenax/charcoal cartridges. The major PICs found were vinyl chloride from 1,2-dichloroethane, 1,1-dichloroethene from 1,1,1-trichloroethane, and carbon tetrachloride from trichloroethene. Chloromethane was also formed at significant levels for all of the compounds incinerated. Additional field samples that were collected and analyzed showed that the precision of the PIC generation was fairly constant. Use of a wide-bore (0.53 mm) capillary column, to improve chromatographic resolution, was also evaluated. This modification was used to enhance the gas chromatography/mass spectrometry performance and allow improved identification of compounds in the field samples.

An Environmental Protection Agency audit cylinder containing compounds at approximately 5 ppb was also sampled and analyzed. Results showed these procedures incorporating VOST methodology were accurate and precise.

The incineration of chlorinated organic compounds is of interest because of their potential health impact. Chlorinated organic compounds can be permanently destroyed by incineration, which in an ideal model produces final combustion products of CO_2, water, and HCl. However, products of incomplete combustion (PICs) are formed during any real waste combustion process. As public concern over incineration of hazardous waste increases, so does the need for better methodology to assess these emissions both qualitatively and quantitatively. The volatile organic sampling train[1] (VOST) method is specified in incineration trial burns as the regulatory method to ensure that the destruction and removal efficiency (DRE) of 99.99 percent is achieved for the principal organic hazardous constituents (POHCs). Normally only two or three organic compounds are selected as POHCs during a trial burn, and the VOST is used to collect information on only these pre-selected compounds. Other hazardous compounds may also be formed as PICs, but these additional compounds would not be identified by the current VOST Protocol.[2] The objective of this study was to evaluate the application of the VOST method and subsequent analytical procedures for the simultaneous measurement of volatile POHCs and PICs present in the incineration exhaust gas stream.

Known concentrations of selected POHCs were incinerated under controlled conditions. A sample of the incinerator exhaust was collected with a multiple simultaneous VOST sampling apparatus by using the SW-846 Method 0030, volatile organic sampling train methodology, followed by analysis using SW-846 Method 5040 (GC/MS), modified by the use of wide-bore (0.53 mm) capillary column chromatography. In addition, a distributive volume sampling study was performed to check for significant breakthrough of PICs. The VOST methodology allows sampling at 0.25, 0.5, and 1 L/min; however, the comparability of sampling at these flowrates has not been evaluated. Distributive volume effects would indicate if a bias could result from sampling at these different flowrates. Effects of flowrate were not significant for this study, so the data were pooled for the estimate of PIC formation.

Literature Search

A literature search on PIC formation from common POHCs was conducted, focusing on several chlorinated organic compounds widely used as POHCs during initial trial burns. These compounds had a potential of forming chlorinated PIC compounds and were considered hazardous to human health. Another objective of the literature search was to recommend for study several POHCs that would produce PICs at levels measurable using the VOST method. The most frequently mentioned POHCs (trichloroethene, 1,1,1-trichloroethane, 1,2-dichloroethane, trichlorofluoroethane [Freon 113]) are suspected of forming PICs (benzene, chloromethane, chlorobenzene, carbon tetrachloride, vinyl chloride, tetrachloroethene, 1,1-dichloroethylene, chloroform). All of these PICs could potentially be formed from any of the listed POHCs during the thermal destruction of chlorinated organics.

Experimental

As an initial step in improving the measurement of PICs in incineration exhaust samples, the analytical portion of the

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VOST method was examined. Figure 1 shows a schematic of the VOST analytical system. In the current methodology for analysis of incinerator samples taken by the VOST, the cartridges are thermally desorbed and the volatilized compounds are purged through water, concentrated on an analytical sorbent trap, and introduced into a gas chromatograph/mass spectrometer (GC/MS) detector from chromatographic separation performed on a 3.0-mm × 2-m glass column packed with 0.1 percent SP-1000 Carbopack B.[3]

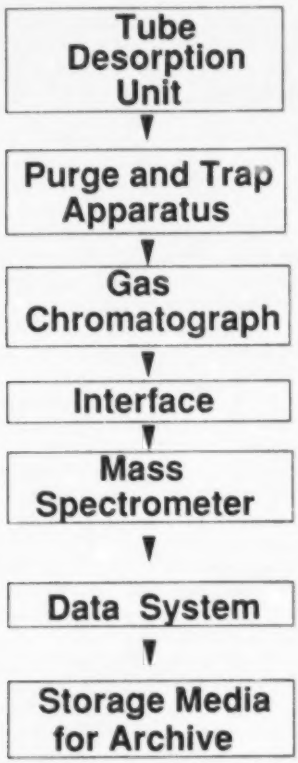

Figure 1. Schematic diagram of the VOST analytical system.

If the sample matrix allows full chromatographic resolution of the compounds of interest, and the concentration of POHCs/PICs does not saturate the chromatographic column or the mass spectrometer detector, then the quantitative analysis of target compounds will be straightforward. A qualitative analysis may also be performed on other chromatographically resolved peaks to obtain an acceptable level of confidence in their identification. As in the case of most real incinerator exhaust gas samples, the sample matrix is a complex mixture with multiple components which coelute and generally cause poor chromatographic resolution. In many cases the level of POHCs/PICs is at or near saturation of the analytical system, producing a high level of background, which when packed column gas chromatography is used gives broad peaks that interfere with the identification of compounds. Interpretation of these spectra is possible only by extensive use of computerized background subtraction techniques coupled with either computerized spectral identification techniques and/or interpretation of the enhanced mass spectrum by a skilled interpreter of mass spectral data; it is a time-consuming process requiring a considerable level of training and experience in mass spectrometric techniques.

The development of wide-bore (0.53 mm) capillary column chromatography provides better chromatographic resolution, and generally a better opportunity to separate these complex samples.[4] Peaks eluted from a capillary column will be narrower (typically 8–14 s wide) than the peaks eluting from a packed column (typically 20–45 s wide). With decreased compound coelution, the quality of mass spectra available for interpretation also improves. The capillary column also provides shorter analysis times while retaining a reasonable sample capacity. The wide-bore (0.53 mm) capillary column has sample capacity approaching that of a packed column while retaining much of the peak resolution of a conventional capillary column. Figure 2 shows the typical analysis of a VOST sample using an SP-1000 packed column, and Figure 3 shows the chromatogram of a field sample desorbed from a Tenax (Alltech Associates, Inc, Avondale, Pa)-Tenax/charcoal cartridge and analyzed by using a wide-bore (0.53 mm) capillary column. An additional benefit of extended compound resolution is also shown in Figure 3. At approximately 28 min, GC/MS compounds such as dichlorobenzene (far less volatile than typical VOST target compounds) are observed. Detection of the less volatile compounds is limited only by the ability to desorb these compounds from the Tenax-Tenax/charcoal sampling cartridge and purge them from water. However, the VOST method may not be acceptable for sampling volatile organics above 130°C.

Two different GC/MS systems were used to evaluate the wide-bore (0.53 mm) capillary column: the Finnigan-MAT 4500 which has a differentially pumped vacuum system with diffusion pumps, and the Finnigan-MAT 5100, which has a turbomolecular pump with no differential pumping. The first configuration consisted of a Megabore (J & W Scientific Inc., Folsom, CA) purge-and-trap concentrator with a DB-624 0.53-mm × 30-m fused-silica capillary column with a 3 μm phase thickness, interfaced directly to the ion source of a Finnigan-MAT 4500 GC/MS/data system. In the second

Table I. Optimum operating parameters for Megabore® VOST analysis on the Finnigan-MAT 4500 and 5100 GC/MS systems.

Cartridge desorb oven	
Desorption gas flow	40 mL/min
Desorb temperature	180°C–200°C
Desorb time	11 min
Carrier gas	Grade 5.0 helium
Tekmar purge-and-trap concentrator	
Analytical Trap	90% Carbotrap® 10% charcoal (w/w)
Purge temperature	Ambient
Purge time	11 min
Desorb temperature	180°C
Desorb time	5 min
Desorb flow	−2 mL/min helium
Gas chromatograph	
Column	DB-624, 0.53-mm ID × 30-m, thick film, fused-silica capillary column
Injection temperature	100°C
Transfer Oven Temperature	240°C
Column Head Pressure	0.14 kg/cm^2
Carrier gas flow	2.3 ml/min helium
Initial temperature/ hold time	5°C/2 min
Program rate	6°C/min
Final temperature/ hold time	240°C/1 min
Mass spectrometer	
Manifold temperature	105°C
Scan rate/range	1.0 s/scan, 35–260 amu
Electron energy	70 eV
Emission current (4500)	0.28 mA
Emission current (5100)	0.50 mA

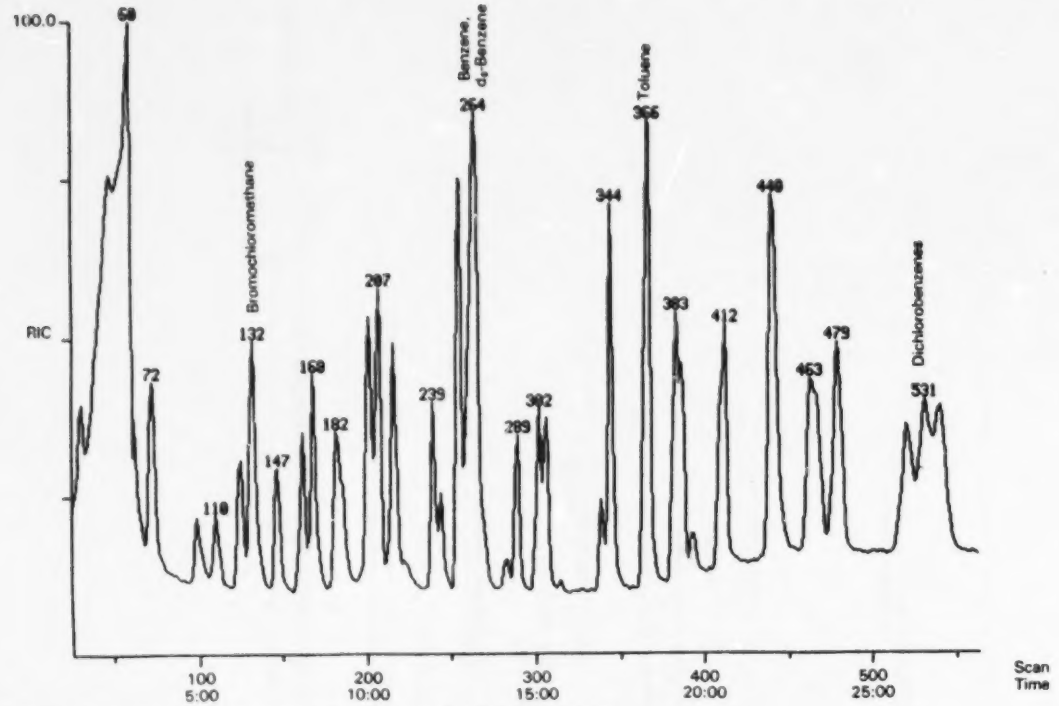

Figure 2. Typical analysis of a VOST sample using an SD-100 packed column.

configuration, a Finnigan-MAT 5100 GC/MS/data system was used with the same column and the same purge-and-trap concentrator. The 5100 system was used to evaluate the wide-bore (0.53 mm) capillary column performance in an MS system which uses a turbomolecular, high-vacuum pumping system without differential pumping between the source and analyzer of the MS. Both of the mass spectrometers use the INCOS data system for data acquisition and processing. The optimum operating parameters for both GC/MS systems are shown in Table I.[4] The only significant change to the current analytical protocol is the use of a 1-min dry purge of the analytical system to reduce the effects of moisture on column resolution. In the dry purge mode, dry carrier gas is passed through the purge-and-trap apparatus for the recommended time, and the flow is then switched so that the purge vessel is bypassed, and for 1 min the carrier gas is flowed directly onto the analytical trap. This dry purge step restores the desired chromatographic peak shape. The evaluation showed both systems to be acceptable.

Tenax-Tenax/charcoal cartridges were prepared in accordance with the current VOST protocol. A clamshell oven tube desorption unit (Figure 1) with 5.0 grade helium carrier gas was used to desorb the VOST cartridges after sampling into a Tekmar LSC purge-and-trap concentrator (LSC-2, Tekmar, Cincinnati, OH) equipped with an analytical trap. The analytical trap was pulse-heated to 190°C and the vola-

Table II. PIC formation (ppb).[a]

PIC	Background, test 1	POHC 1,2-Dichloroethane, test 2	POHC 1,1,1-Trichloroethane, test 3	POHC Trichloroethene, test 4
Vinyl chloride	ND	>300 (S)[b]	ND	ND
Chloromethane	1.0	90	>200 (S)	>200 (S)
Tetrachloroethene	ND	6.5	0.3	12
Methylene chloride	ND	4.3	3.6	ND
Trichloroethene	ND	1.0	2.7	>100 (S)
1,1-Dichloroethene	ND	1.1	>200 (S)	26
Carbon tetrachloride	ND	0.1	6.6	>60 (S)
Chloroform	ND	0.3	3.3	0.7
1,1,1-Trichloroethane	ND	0.1	0.4	0.2
t-1,2-Dichloroethene	ND	0.3	1.0	ND
m-Dichlorobenzene	ND	0.4		ND
p-Dichlorobenzene	ND	0.2	ND	ND
o-Dichlorobenbzene	ND	0.2	ND	ND
1,2-Dichloroethane	ND	0.4	ND	ND

[a] Sampling was conducted at 1 L/min for 20 min on Tenax/Tenax-Charcoal tubes. All values are averages of four samples.
[b] (S) = saturated GC/MS detector.
ND = not detected

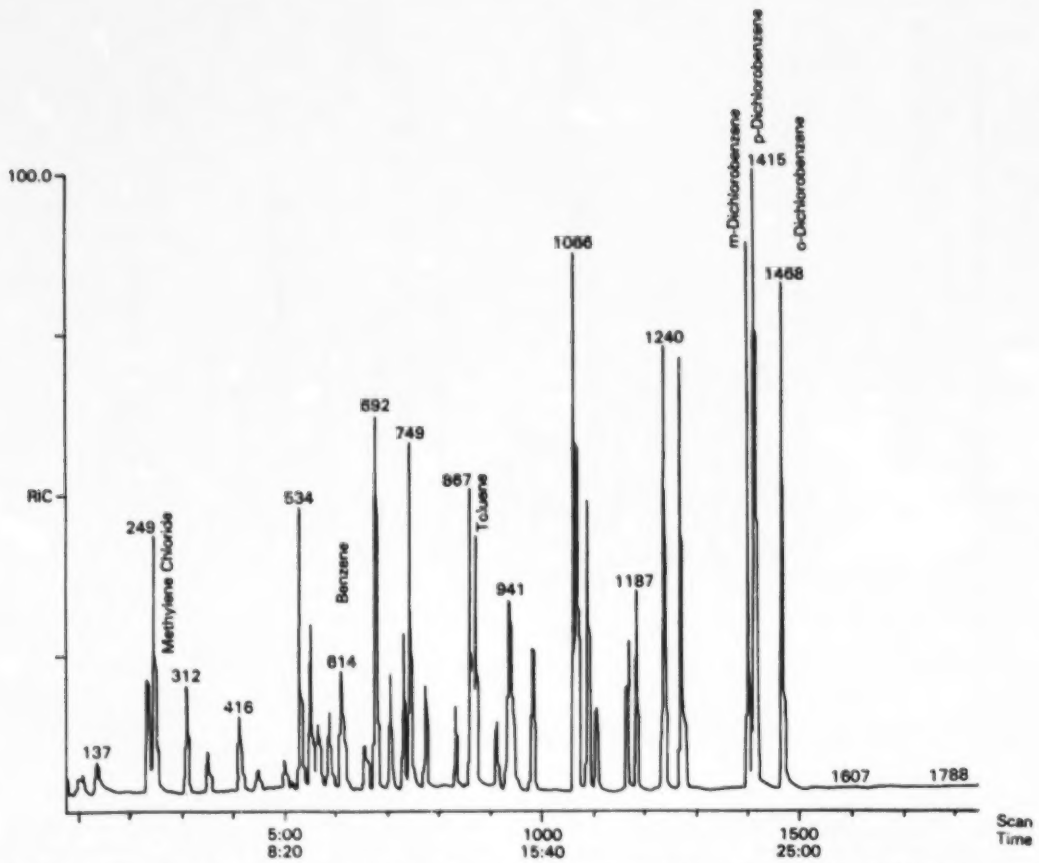

Figure 3. Chromatogram of a field sample desorbed from Tenax/charcoal cartridge and analyzed by using the Megabore column.

tile organics were swept onto the DB-624 wide-bore (0.53 mm) capillary column with helium carrier gas. Calibration standards were prepared from EPA Method 624 purgeable standards (Supelco Corporation, Bellefonte, PA, and Ultra Scientific, Hope, RI).[1] These standards were spiked on the VOST tubes and were analyzed several times to maximize instrument sensitivity and resolution.

Field Measurements Program

To show that these analytical changes would improve the chromatographic resolution of the VOST analytical procedure and allow for a more accurate characterization of both POHCs and PICs, authentic field samples which contained significant levels of PICs were collected. Because well-oper-

Table III. PICs formed from incineration of 1,1,1-trichloroethane in tests 3, 5 and 6.

PIC	Test 3[a]		Test 5[b]		Test 6[c]	
	1 L/min 20 Liters				0.5 L/min 10 Liters	
		%CV[d]		%CV[d]		%CV[d]
Vinyl chloride	ND[e]		ND		ND	
Chloromethane	>200(S) ppb		38 ppb		14 ppb	
Tetrachloroethene	0.3	60	14	18	0.1	54
Methylene chloride	3.6	22	1.1	19	0.8	4
Trichloroethene	2.7	24	7.8	12	1.9	44
1,1-Dichloroethene	>200(S)		>200(S)		27	
Carbon tetrachloride	6.6	27	5.0	4	3.3	40
Chloroform	3.3	7	2.1	27	2.1	41
1,1,1-Trichloroethane	ND		ND		ND	
t-1,2-Dichloroethene	1.0	19	0.3	24	0.4	10
Average %CV		28%		17%		37%

[a] Test 3 is an average of four simultaneous VOST trains.
[b] Test 5 is an average of three simultaneous VOST trains.
[c] Test 6 is an average of two simultaneous VOST trains.
[d] %CV calculations did not include saturated compound (S).
[e] ND = not detected; (s) = saturated GC/MS detector.

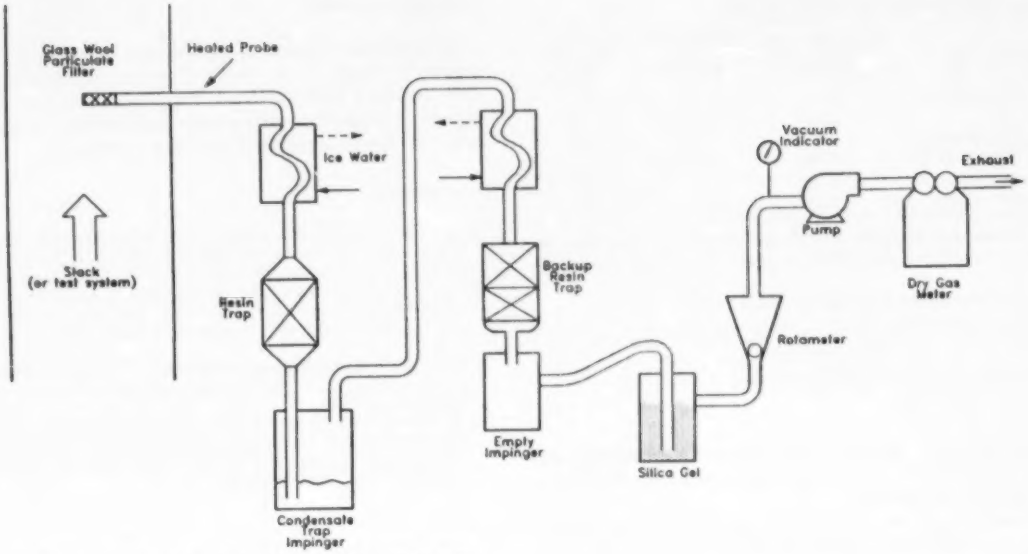

Figure 4. Schematic of single volatile organic sampling train.

ated and permitted incinerators cannot always run in a mode where measurable PIC levels are present, it was necessary to conduct these tests on a small pilot-scale incinerator which could be fed stable, known levels of POHCs under controlled operating conditions. A Resource Conservation and Recovery Act Research and Development and Demonstration permit was obtained for this purpose. A pilot-scale incinerator was leased from ARI International. This incinerator was a new model CTP 500 (dual-fuel) pilot unit and was used as a direct thermal oxidizer without any catalyst. Normal operating temperatures were maintained between 1200°F (649°C) and 1400°F (760°C). The exhaust flow of the incinerator was approximately 500 standard cubic feet per minute. These specifications were suitable for the objectives of the program and were not intended to be representative of current full-scale incinerators in operation. The POHCs were injected into the incinerator burner on a continuous basis in the gas phase. The individual POHC inlet concentrations were measured during each test by GC/flame ionization detection (FID) by a Shimadzu Mini-2 GC/FID. Several POHC concentration measurements were made during each test to verify that the feed composition remained constant during the program. The carbon monoxide levels were monitored continuously during each test with a Bendix IR analyzer as an indicator of combustion control and process stability.

All sampling was done in simultaneous pairs or quadruplicate runs at either 0.25, 0.5, or 1 L/min for 20 min using the VOST apparatus shown in Figure 4. A standard pair of VOST tubes was used.

Results of Field Tests

The objective of the first set of tests in which the selected POHCs were incinerated separately was to determine which PICs were formed in sufficient quantity to be detected and quantified by the VOST method. The sampling apparatus was used to collect simultaneous samples with the VOST so that average PIC concentration could be used in the selection of the optimum POHC. Test 1 was conducted as a background test with the incinerator in a normal operation mode with no POHC present. Tests 2, 3, and 4 were conducted with separate combustion of the POHCs 1,2-dichloroethane, 1,1,1-trichloroethane, and trichloroethene respectively. Table II summarizes this series of tests. The combustion of 1,2-dichloroethane (test 2) had a high DRE (99.999 percent) and also produced many PICs but was unacceptable for further study as a test POHC because of the potential release of vinyl chloride at levels that were greater than 300 ppb. The combustion of trichloroethene (test 4) had a lower DRE (99.9 percent) but formed fewer PICs; trichloroethene was not used in any further tests. The combustion of 1,1,1-trichloroethane (test 3) was characterized by a high DRE (99.999 percent) with no detectable levels of vinyl chloride; however, several other PICs were formed at sufficient but low part-per-billion levels that 1,1,1-trichloroethane was useful as a POHC for additional PIC experiments.

In addition to the PIC compounds quantified in Table III, several other trace level compounds were identified by GC/MS analysis, including chloroacetylene, benzene, C_8H_{18}, bromochloropropane, chlorobenzene, 1,1,1,2-tetrachloroethane, and benzaldehyde (benzaldehyde may be a Tenax decomposition product).

After the first series of tests were analyzed and 1,1,1-trichloroethane was selected as the test POHC, minor operating modifications to the incinerator were made, and test 5 was conducted to confirm that PIC formation was unchanged. The series of tests showed the PIC formation consistent with previous results. In all of the field samples, the combustion conditions were maintained as closely as possible to the initially determined conditions. The data from the 14 VOST tube pairs are summarized in Table IV. Although these samples were taken over several weeks of incinerator

Table IV. Test 5 results: PICs detected over several weeks with 14 VOST tube pairs.

Compound	Average conc. (ppb)[a]	Coeff. of variation
Vinyl chloride	ND	—
Chloromethane	(S)	—
Tetrachloroethene	3.8	45
Methylene chloride	6.0	13
Trichloroethene	3.4	17
1,1-Dichloroethene	27.0	—
Carbon tetrachloride	5.2	17
Chloroform	3.4	20
1,1,1-Trichloroethane	ND	—
1,2-Dichloroethene	1.2	18

[a] ND = not detected; (S) = saturated GC/MS detector.

Table V. Quality assurance audit results.[a]

Compound	Reported value mean (ppb)	Precision[b] percent	Accuracy[c] percent
Methylene chloride	6.35	6.0	27.
Chloroform	5.43	3.3	8.6
1,1,1-Trichloroethane	6.07	1.6	30.
Carbon tetrachloride	7.04	2.0	50.
Trichloroethene	6.22	1.8	11.
Tetrachloroethene	3.76	0.27	−25.

[a] Audit cylinder was sampled at 0.5 L/min for 20 min. Analysis was performed according to the VOST protocol (Megabore® modification).

[b] Precision % = (standard deviation/mean) * 100.

[c] Accuracy % = $\frac{\text{(reported value − accepted value)}}{\text{accepted value}} * 100$.

Accepted value = cylinder calibration value.

operation, the PIC formation was extremely consistent, as shown by a coefficient of variation generally below 20 percent for all test compounds.

As a final check on accuracy of the PIC measurement procedure, an audit was conducted using a cylinder gas mixture of known part-per-billion concentration. The data from two simultaneous audits are shown in Table V. Only six compounds of the 18-component mixture were substances for which the GC/MS was calibrated. Nevertheless, except for two compounds (vinyl chloride and 1,3-butadiene) 18 compounds were identified by the GC/MS analysis procedure. Using the d_6 benzene standard, an estimated concentration for these additional compounds was calculated; all were generally within a factor of 2 of the true value.

Conclusions

As a result of this study, the use of wide-bore (0.53 mm) capillary columns in VOST analysis was shown to improve the resolution of GC/MS analysis in the identification and quantification of PICs and improved the resolution of POHCs in the DRE calculations.

Acknowledgment

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